

[Second Edition.]

PATENT SPECIFICATION



Convention Date (Germany) : June 1, 1935.

475,882

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(Patent of Addition to No. 466,042 : Dated Nov. 24, 1934.)

Complete Specification Accepted : Nov. 29, 1937.

COMPLETE SPECIFICATION.

Process for the Manufacture of Highly Dispersed Pigments.

We, I. G. FARBENINDUSTRIE AKTIEN-
GESELLSCHAFT, a joint stock company
organised under the laws of Germany,
of Frankfort-on-Main, Germany, do hereby
5 declare the nature of this invention and in
what manner the same is to be performed,
to be particularly described and ascer-
tained in and by the following statement :—

This invention relates to the manufac-
ture of highly dispersed pigments and is a
modification of that of specification No.
32476/35. (Serial No. 466,042).

Specification No. 32476/35 (Serial No.
466,042), describes and claims composi-
tions comprising metal containing phthalocyanines and dispersing agents, which
compositions are obtained by mixing or
milling metal-containing phthalocyanines
with dispersing agents in the presence of
water.

In accordance with the present invention
metal free phthalocyanines can likewise
be improved as regards softness of grain,
besides clarity and intensity of shades,
25 whereby similar advantages in practical
application accrue by subjecting the same
to a treatment with water soluble dispersing
agents in the presence of water. This
treatment can be effected at elevated
30 temperature and is suitably carried out in a
closed vessel, in which case the pressure will
usually be above atmospheric. The degree
of dispersion depends on the duration of the
dispersing treatment, on the choice of the
35 dispersing agents and on the temperature
applied. The powdered dyestuffs obtained
from these dispersions by evaporation are
as stated above distinguished by their
good softness of grain. The dyestuff may
40 also be filtered with suction from the
solution in order to remove a part
of the dispersing agent. Prior to the dis-
persing treatment the metal-free phthalocyanines
are preferably pasted by dissolving
45 the same in suitable acids, such as
concentrated sulphuric acid, chlorsulphonic
acid or trichlorsulphonic acid with sub-
sequent precipitation by means of water.

As dispersing agents such compounds
50 are preferably employed as have at the
same time a wetting capacity. Reference
is made to the examples given in the

Specification of the parent patent.

The softening of the grain of the metal
free phthalocyanines and their conversion
into a finely divided state by means of
dispersing agents can be effected merely
by intensively mixing or stirring these
compounds in the presence of water, if
desired at an elevated temperature, or by
working in a ball mill or similar device.
The preparations produced by the methods
described above may be applied for the
colouring of artificial masses, particularly
for addition to spinning solutions to be
used for the production of artificial silk.

The following examples illustrate the
invention, the parts being by weight :—

EXAMPLE 1.

50 parts of the metal-free phthalocyanine
obtained by heating ortho-phthalonitrile or
4-chlorophthalonitrile in the presence
of formamide, are dissolved in 400 parts
of concentrated sulphuric acid, poured onto
ice, filtered and then rinsed. The paste
thus obtained is diluted with water and
boiled for 1 hour after the addition of 1
part of Turkey red oil. After separating
and drying a loose, well distributable
greenish-blue dyestuff powder is obtained
which is especially suitable for the colouring
of lacquers and for printing pastes.

EXAMPLE 2.

50 parts of the metal-free phthalocyanine
as described in the preceding example are
dissolved in concentrated sulphuric acid.
The solution is then poured onto ice and
the precipitated dyestuff is filtered with
suction. The phthalocyanine obtained is
suspended in water and boiled and the
paste obtained is milled in the ball or
colloid mill with 2.5 parts of the sodium
salt of a condensation product of formalde-
hyde and β -naphthalene sulphonate acid.
The paste thus obtained is particularly
suitable as wall-paper colour on account of
its excellent strength.

The condensation product of formalde-
hyde and β -naphthalene sulphonate acid
referred to in the preceding paragraph 100
may be prepared by adding 4 parts of a
30% formaldehyde solution at about 60
to 100° C. to the crude mixture contain-
ing β -naphthalene sulphonate acid obtained

by heating 10 parts of naphthalene with 12.5 parts of a 98% sulphuric acid to about 130—140° C. The whole is then heated while stirring until free formaldehyde can no longer be traced.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is :—

- 10 1. Pigment compositions comprising metal-free phthalocyanines and dispersing agents which compositions are obtained by mixing or milling metal-free phthalocyanines with water-soluble dispersing agents in the presence of water.
- 15 2. Compositions as claimed in claim 1 in the production of which the mixing or

milling is effected at an elevated temperature and/or pressure.

3. Compositions as claimed in either of the preceding claims from which a part of the liquid has been removed.

4. The compositions substantially as described in the examples.

5. The application of the preparations claimed in any of the preceding claims for the colouring of artificial masses, particularly for addition to spinning solutions to be used for the production of

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Dated this 27th day of May, 1936.
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W.C.2.

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